

Comparison of Denture Base Material Processed by the Traditional and a Two-cycle Method

Undergraduate Research Thesis

Presented in partial fulfillment of the requirements for graduation *with
honors research distinction* in Biochemistry in the undergraduate
colleges of The Ohio State University.

By

Gefei Wang

The Ohio State University

Project Advisor: Professor Fengyuan Zheng, College of Dentistry
Professor Scott Schricker, College of Dentistry

Defense Committee: Dr. Scott Schricker, Dr. John Shimko, Dr.
Fengyuan Zheng and Dr. Richard Swenson

ACKNOWLEDGEMENTS

First, I want to acknowledge and thank Dr. Fengyuan Zheng, for introducing me to this project and for his guidance and encouragement. To my honors advisor, Dr. Scott Schricker, thank you for your helpfulness and willingness to answer all of my questions throughout the past two years. I would like to thank Dr. Mostafa Ibrahim for all of his help making the specimen for the study. I want to thank the lab technician, Mr. Carl Kipp, for teaching me the proper methods of using the lab equipment. I would like to thank Dr. Callam Christopher for allowing me to use equipment from organic chemistry lab. I would also like to thank Dr. William Johnston for helping me measure the color and transperence of the specimen. I would like to thank Dr. John Shimko and Dr. Richard Swenson for serving as my defense committee members. I am grateful to the financial support from ASC Undergraduate Research Scholarship and Rudy Melfi Fellowship. Finally, I would like to thank my family for always loving and supporting me in everything that I do.

ABSTRACT

Poly(methyl methacrylate) PMMA is the preferred polymer to fabricate denture base materials. However, the current processing conditions results in unpolymerized methyl methacrylate in the final denture base. This can result in lower mechanical and physical properties and lead to biocompatibility issues. Changing the processing method will change the properties of the denture base materials. Recently, the traditional processing method has been improved. The commonly used processing technique is a one cycle heat-curing: the PMMA resin is heated gradually to 75°C and remains at that temperature for 9 hours. In the new method, after initial heating, the denture base is cooled down to room temperature followed by an additional processing step at a temperature of 120°C and at a pressurized water bath. The degree to which the novel two-cycle method would change the properties of the denture base material remains unknown. The object of this study is to compare the physical properties of materials processed by two methods and assess the effectiveness of the two-cycle method. In this study, the control group is processed by the conventional one-cycle method and the experimental group was processed using the two-cycle method. The hardness, color stability, solubility in water and flexural strength were measured. It is shown that the two-cycle processed denture base is harder and less soluble than the one-cycle processed denture base. This suggests that denture base materials with improved mechanical properties can be manufactured via the two-cycle method. Between the two groups, there was no statistically significant difference for either color stability or flexural strength. The overall result suggests that we can improve some properties of denture base material by using two-cycle method, which is environmentally friendly and low-cost.

Table of Contents

Acknowledgement	1
Abstract	2
Table of Content	4
1.Introduction	
1.1 Current issue	4
1.2 Enhancing the quality of PMMA material	5
1.3 Hypothesis.....	6
1.4 Hardness	6
1.5 Solubility.....	7
1.6 Color stability	8
1.7 Flexural strength.....	10
2. Methodology	
2.1 Materials	12
2.2 Sample preparation.....	12
2.3 Hardness	13
2.4 Solubility.....	14
2.5 Flexural strength.....	14
2.6 Color stability	16
3.Results	
3.1 Hardness	17
3.2 Solubility.....	18
3.3 Flexural strength.....	19
3.4 Color stability	20
4. Discussion.....	22
5. Conclusion	25
6. Recommendation	26
7.Appendix	27

1 Introduction

1.1 Current issue

Poly(methyl methacrylate) PMMA is the preferred polymeric material to fabricate denture bases¹ due to mechanical and physical properties that match the clinical requirements. It is an ideal material for the manufacture of denture bases because of the simple manufacturing process, low cost, light weight and color which match the color of gum tissues². However, compared to other dental materials, PMMA's hardness, flexural strength, and ability to resist aging are relatively low². The PMMA denture base manufactured with the conventional method has a high risk of breakage. Vojdani *et al.*² found that 68% of PMMA dentures have fractures within a few years after manufacture. One study showed that 36% of patient stopped using their dentures after one year of insertion³ while other studies suggested a higher percent of denture retention⁴. Fractures and wear of PMMA happen frequently and repairs are difficult. Moreover, denture base materials have a potential to cause adverse reactions because of the solubility of unpolymerized material and biofilm formation⁵. Because the dentures have direct contact with the gum tissue, the gums are risk of fungal infections and allergic reactions⁶. Finally, the aesthetics of the dentures could be affected by colored beverages⁷.

¹ Hassain S. Textbook of dental materials. India: Jaypee Brothers Medical Publishers (P) Ltd. (2004).

² Vojdani M, Bagheri R, Khaledi AAR. Effect of aluminium oxide addition on the exural strength, surface hardness, and roughness of heat-polymerized acrylic resin. *J. Dent. Sci.* **7**, (2012) 238-244

³ Akeel R. Usage of removable partial dentures in Saudi male patients after 1 year telephone interview. *Saudi Dent J.* 2010;22:125–128.

⁴ Rehmann P, Orbach K, Ferger P, et al. Treatment outcomes with removable partial dentures: a retrospective analysis. *Int J Prosthodont.* 2013;26:147–150.

⁵ Weaver RE, Goebel WM *J Prosthet Dent.* 1980 Feb; 43(2):138-42.

⁶ de Freitas Fernandes FS, Pereira-Cenci T, da Silva WJ, et al. Efficacy of denture cleansers on *Candida* spp. biofilm formed on polyamide and polymethyl methacrylate resins. *J Prosthet Dent.* 2011;105:51–58.

⁷ Imirzalioglu P, Karacaer O, Yilmaz B, Ozmen I. Color stability of denture acrylic resins and a soft lining material against tea, coffee, and nicotine. *J Prosthodont* 2010;19:118-24.

1.2 Enhancing the quality of PMMA material

There are many ways to improve the mechanical properties of the PMMA dental resins. Glass fiber, a cost-effective inert material with high modulus, is a good candidate for the mechanical reinforcement of PMMA. But the glass fibers need to be silanized to activate the glass fiber's surface⁸. Polyamide fiber, including Nylon and Aramid, is also used to enhance the flexural strength and flexural modulus, though high content of polyamide fiber compromises the hardness of the denture base⁹. Polyethylene fiber bolsters the elastic modulus and the impact strength of denture base material¹⁰. Adding filler is another common way to strengthen the PMMA material. Alumina, zirconia and titanium dioxide are common fillers; in addition metal oxide, nano-carbon, mineral and noble metal(such as silver) can also be used as fillers¹¹.

The quality of denture base materials could also be improved by modifying the processing technique. Changing temperature cycles and polymerization time can influence the material properties. A study has shown that longer polymerization time leads to longer polymer chains in the denture base and reduced residual monomer content¹². Changing the processing method will change the properties of the denture base materials. Recently, the traditional processing method has been improved by Dr. Zheng from College of Dentistry at OSU. The commonly used processing technique is a one cycle heat-curing: the PMMA resin is heated gradually to 75°C and remains at that temperature for 9 hours. In the new method, after initial

⁸ Moreno-Maldonado V. Journal of applied polymer science: Fiber-reinforced nanopigmented poly(methyl methacrylate) as improved denture base. John Wiley & Sons Inc; 10/2012;126:289.

⁹ Gad MM. International journal of nanomedicine: PMMA denture base material enhancement: a review of fiber, filler, and nanofiller addition. Dove Medical Press; 2017;12:3801.

¹⁰ Uzun G, Hersek N, Tinçer T. Effect of five woven fiber reinforcements on the impact and transverse strength of a denture base resin. J Prosthet Dent. 1999;81:616–620.

¹¹ Gad MM. International journal of nanomedicine: PMMA denture base material enhancement: a review of fiber, filler, and nanofiller addition. Dove Medical Press; 2017;12:3801.

¹² Wong DM, Cheng LY, Chow TW, Clark RK. Effect of processing method on the dimensional accuracy and water sorption of acrylic resin dentures. J Prosthet Dent 1999;81:300-4.

heating, the denture base is cooled down to room temperature followed by an additional processing step at a temperature of 120°C and at a pressure of 15psi. The aim of the second cycle is to increase the degree of polymerization which should lead to improved physical and biochemical properties. However, the degree to which the two-cycle method will change the denture base material remains unknown.

1.3 Hypothesis

The main purpose of this research is to compare the differences in the physical properties (hardness, color stability, flexural strength, solubility) of materials processed by the conventional method and the new method. The first null hypothesis was that a PMMA denture base, processed by the new method, would have equal hardness as the denture resin processed by the traditional method. The second null hypothesis was that the processing technique would not affect solubility of the PMMA materials. The third null hypothesis was that processing technique would not affect the color stability of the materials. The fourth null hypothesis was that the flexural strength of the PMMA resin remains the same even though the processing method changed.

1.4 Hardness

Hardness is a property that provides denture base with resistance to forces in oral cavity, scratching and abrasion¹³ and is directly related to morphology, chemistry and polymerization¹⁴. Based on the definition of hardness, resistance to the indentation, the common way to determine the hardness is to measure the depth of the indentation. Vickers, Knoop, Brinell, Shore and Rockwell are the commonly used hardness test methods¹⁵. These hardness tests are differentiated

¹³ Mansour M, Wagner W, Chu TM. Effect of mica reinforcement on the flexural strength and microhardness of poly- methyl methacrylate denture resin. J Prosthodont. 2013;22(3): 179-183.

¹⁴ Parr GR, Rueggeberg FA. In vitro hardness, water sorp on, and resin solubility of laboratory-processed and autopolymerized long-term resilient denture liners over one year of water storage. J Prosthet Dent. 2002;88(2):139-144.

¹⁵ Germak A, Herrmann K, Low S. Traceability in hardness mea- surements: from the de ni on to industry. Metrologia. 2010;47(2):2.

by the shape and dimension of their indenters. Vickers and Knoop hardness tests are the most commonly used methods for thermoplastic denture base polymers because of their relative low hardness¹⁶. A load is applied to the sample surface for a given amount of time. In Vickers test, both diagonals of the indentation are measured, and the average value is used, while in the Knoop test, only the longer diagonal is measured. To get more accurate data, Vickers hardness test method was selected for this study. The equation is: $HV = \frac{2F \sin \frac{136^\circ}{2}}{d^2} \approx 1.854 \frac{F}{d^2}$, where F is the load, d is the diagonal length of Viker indenter.

1.5 Solubility

After polymerization, the unpolymerized portion of denture resin is trapped between long chains of polymers. When the denture is immersed in water, the unpolymerized monomers leach out in water. Therefore, the room previously occupied by those monomers replaced by water and the physical properties of the structure will be compromised. The monomers could also trigger soft tissue reaction. According to a study, as the degree of polymerization increases, the solubility of the denture decreases¹⁷. Solubility in water, thus can be used to indirectly compare the degree of plasticization in PMMA. The temperature in oral cavity varies because of eating and drinking cold and hot objects or liquids, which can have a negative effect on the composition of the material and can thus change the physical properties of the material¹⁸. Therefore, a dental base should be tested in conditions similar to those they will be encounter in the intraoral

¹⁶ Ke YL, Dong FX. Hardness of materials: studies at levels from atoms to crystals. *Chin Sci Bull.* 2009;54(1):131-136.

¹⁷ Tuna SH, Keyf F, Gumus HO, Uzun C. The Evaluation of Water Sorption/Solubility on Various Acrylic Resins. *European Journal of Dentistry.* 2008;2:191-197.

¹⁸ Longman CM, Pearson CJ. Variation in temperature of the oral cavity during the imbibition of hot and cold fluids [special issue]. *J Dent Res.* 1984;63:521.

environment¹⁹. A thermal cycling procedure is a common method for examining the long-term use of dental materials. The temperature range in the oral cavity is between 5°C- 55°C, therefore the temperature for hot and cold water bath in thermocycling is 55°C and 5°C, respectively¹³. The time length of each bath is 1 min, which is similar to the duration of beverage in mouth. The formula is *relative mass loss* = $\frac{m_0 - m_f}{m_0}$, where m_0 is the initial mass, m_f is the mass of the sample after thermal cycling and dehydration.

1.6 Color stability

Dental base materials are a substitute of the gum and are in directly contact with the oral soft tissue. Therefore, denture base materials should have good color stability and translucency under clinical conditions. Staining of the denture could be caused by exposure to colored beverages²⁰. The degree of staining varies based on the physical and chemical properties of the materials. Degree of polymerization, surface roughness, water absorption and solubility, hydrophilicity are examples of properties that are related to the color stability²¹. Therefore, it is of importance to measure the color change of the acrylic resin after immersing in several beverages. Tea, coffee, wine and artificial color are known for causing discoloration²². In this study, black tea (Lipton), coffee (Folger), and coke (Coca-cola) were chosen as the medium. The color change of the material is measured using gray backing and calculated based on CIEDE2000 standard with K_L , K_C , and K_H equaled to 1. The formula is a modification of the

¹⁹ Ayaz, Elif Aydoğan, et al. "Effects of Thermal Cycling on Surface Roughness, Hardness and Flexural Strength of Polymethylmethacrylate and Polyamide Denture Base Resins." *Journal of Applied Biomaterials & Functional Materials*, vol. 13, no. 3, Sept. 2015, pp. e280-e286. EBSCOhost, doi:10.5301/jabfm.5000236.

²⁰ Imirzalioglu P, Karacaer O, Yilmaz B, Ozmen Msc I. Color stability of denture acrylic resins and a soft lining material against tea, coffee, and nicotine. *J Prosthodont* 2010;19:118-24

²¹ Lai YL, Lui HF, Lee SY. In vitro color stability, stain resistance, and water sorption of four removable gingival flange materials. *J Prosthet Dent* 2003;90:293-300.

²² Makhija PP, Shigli K, Awinashe V. Evaluating the efficacy of denture cleansing materials in removal of tea and turmeric stains: An *in vitro* study. *Indian J Dent Res* 2016;27:528-34

original formula: $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$ where L^* indicate lightness, $\Delta L = L_t - L_0$, a^* indicate redness to greenness, $\Delta a = a_t - a_0$, b indicate yellowness to blueness, $\Delta b = b_t - b_0$ (L_t , a_t , b_t : values at the time of measurement after immersion / L_0 , a_0 , b_0 : values before immersion)²³.

Relative translucency parameter for the specimens was calculated using the formula:

$$RTP_{CIEDE2000} = [(L_{white*} - L_{black*})^2 + (a_{white*} - a_{black*})^2 + (b_{white*} - b_{black*})^2]^{1/2}.$$

1.7 Flexural strength

Flexural strength is a compound property that incorporates aspects of compression and tension. As a foundation, for a denture or prosthesis, the acrylic resin materials should exhibit a high resistance to plastic deformation²⁴. Flexural failure is one of the most common issues for denture base materials. Catastrophic failure happens when a heavy load exceeds the mechanical capacity of the denture base material. Thus, it is necessary to conduct the flexural strength test; it also sheds light on the modulus which is useful in comparing the denture base materials²⁵. In this study, two sample geometries were utilized. One has a geometry that complies with ISO standard (65mm×2mm×10mm) (Figure 1.2), the other group of specimen has a geometry that mimics the clinical denture base material (Figure 1.1). Both were studied since they reflected the load arrangement in a clinical situation. The formula for flexural strength is $FS = 3Pl/2bd^2$ with P being the load at the fracture of the sample, l being the distance between the supports, b being the width of the sample, and d being the thickness of the sample. In this experiment, the dimensions

²³ Jang D-E, Lee J-Y, Jang H-S, Lee J-J, Son M-K. Color stability, water sorption and cytotoxicity of thermoplastic acrylic resin for non metal clasp denture. *The Journal of Advanced Prosthodontics*. 2015;7(4):278-287. doi:10.4047/jap.2015.7.4.278.

²⁴ Diaz-Arnold AM. The Journal of prosthetic dentistry: Flexural and fatigue strengths of denture base resin. C.V. Mosby Co; 07/2008;100:47.

²⁵ Doğan O, Bolayir G, Keskin S, Dogğan A, Bülent B. The evaluation of some flexural properties of a denture base resin reinforced with various aesthetic fibers. *Journal Of Materials Science: Materials In Medicine* [serial online]. June 2008;19(6):2343-2349. Available from: Computers & Applied Sciences Complete, Ipswich, MA. Accessed January 3, 2018.

of the flat sample are: $l = 50.0\text{mm}$, $b = 10.0\text{mm}$, and $d = 3.0\text{mm}$. For curved sample, the distance between two ends is 65mm, and $l = 50.0\text{mm}$, $b \approx 20.0\text{mm}$, and $d \approx 2.6\text{mm}$.

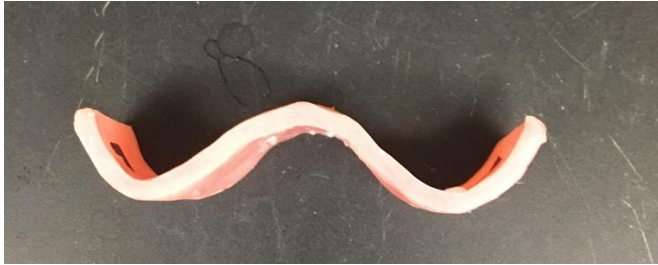


Figure 1.1 Curved flexural strength sample

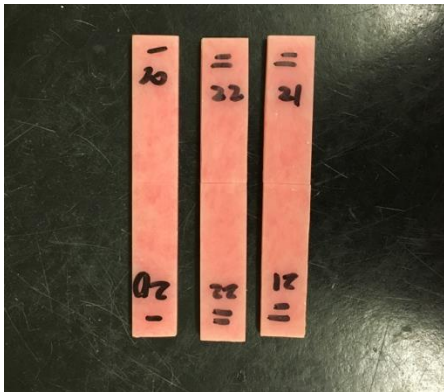


Figure 1.2 Rectangular flexural strength sample

2. Methodology

2.1 Materials

The materials used in this study are Buff stone (ISO type 3 stone; Whip mix), Mounting plaster (ISO type 1 stone; Whip mix), poly(methyl methacrylate)(Luciton 199; Dentsply). The flask used is ejector type flask (Figure 2.1) (Atlas; Handler MFG. Co. Inc.). The processors include the denture curing unit (2010DCU; Handler MFG. Co. Inc.)(Figure 2.2A) and 8.5 qt. stainless steel oval pressure cooker (Big Boss)(Figure 2.2B).



Figure 2.1 Flask



A

B

Figure 2.2 Denture curing unit(A), Pressure cooker(B)

2.2 Sample Preparation

The prototype of the of the specimen were made from steel (Figure 2.3A). Based on the mold, the two-piece casts were made from type III stone, whose powder: water ratio is 1:30 in mass (Figure 2.3B). Then the cast was sealed with wax and put in the flask. Mounting plaster was used in flasking, the process to fill the space between flask and the cast (Figure 2.3C). A

commercially available, heat-polymerised acrylic denture base resin (Lucitone 199) was used in this study. The powder portion was mixed with the liquid portion and the ratio is 3:1 in volume. After mixing, the resin was allowed to sit for 5-10 min before being placed between the casts in the flask. The flask was compressed three times using a flask press (Figure 2.3D). The extra materials were removed using a razor blade between each compression. After compression, the flasks were clamped tightly and then put into the water-filled processor. The flasks were then heated slowly to 95°C during a 9-hour process. Then the control group specimen were taken out of the flask. After the heating cycle, the specimen designated for two-cycle method were then allowed to cool down to the room temperature and heated under 15 psi for about 55 min using a pressure cooker (Big Boss). Specimen with visible air bubbles inside were disused. All of the specimen were sanded manually and stored in distilled water at 37°C before testing.



A

B

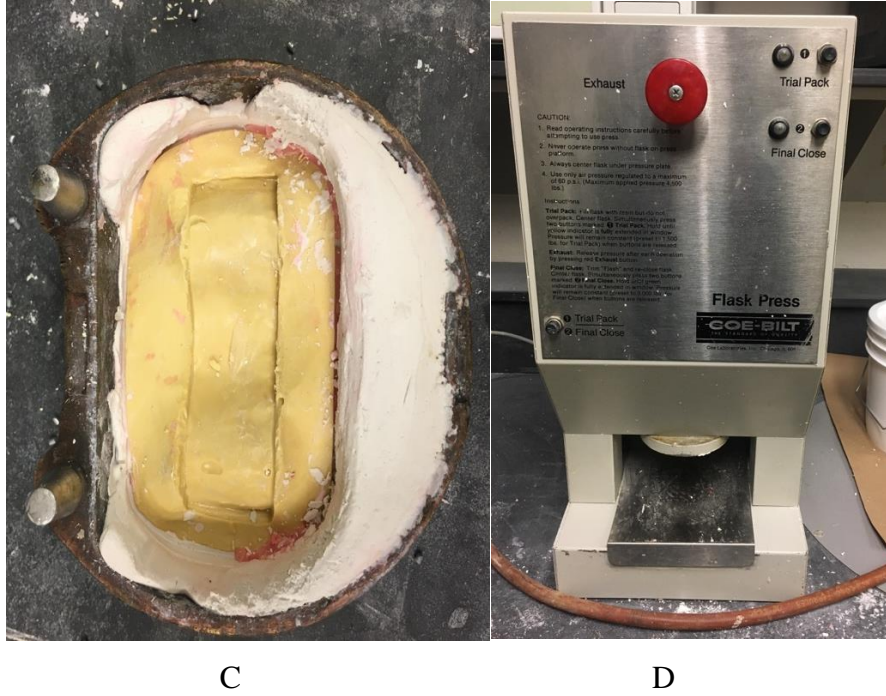


Figure 2.3 From left to right, first row: (A) steel prototype, (B) cast (buff stone), second row: (C) flasking, (D) flask press

2.3 Hardness

The specimen were shaped into round discs with a 10 mm diameter and a 2 mm thickness. Two groups (control group and experimental group) were prepared for the experiment with 5 specimen in each group. The specimen were polished using sand paper(P4000) to obtain a smooth surface. Then the hardness was determined by Vicker hardness test. The equation is:

$$HV = \frac{2F \sin \frac{136^\circ}{2}}{d^2} \approx 1.854 \frac{F}{d^2}$$

Where F is the load, d is the side length of Vicker indenter. The specimen were polished using sand paper(P4000) to obtain smooth surface. To determine Vickers values, a load of 100g was applied for 30 seconds on the specimens using a square microhardness tester.

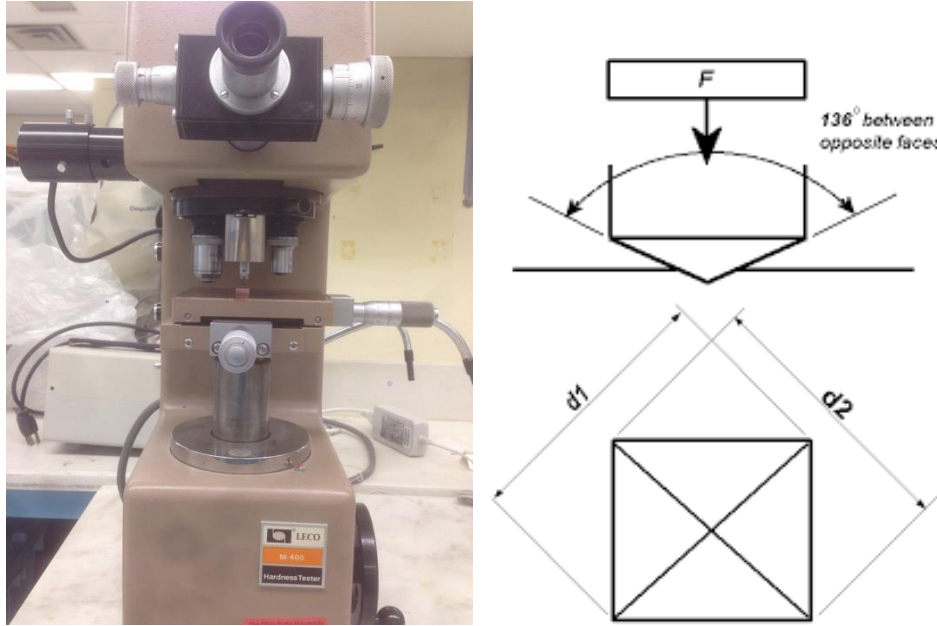


Figure 2.4 Viker hardness tester(left), shape of the indenter²⁶(right).

2.4 Mass loss in water

The specimen were shaped into round disc with 10 mm diameter and 2 mm thickness. All specimen were stored in the desiccator for 48 hours before being weighed. Then, the specimen were put in thermal cycling apparatus (55°C hot bath and 5°C cold bath) for 3000 cycles (1cycle=1min in the hot bath and 1 min in cold bath), this process mimics the aging of the material. After thermal cycling, the specimen were stored in the desiccator for a week, then the mass of the specimen was measured again. The formula is *relative mass loss* $= \frac{m_0 - m_f}{m_0}$, where

²⁶ *Vickers Hardness Test*.;date unknown. Available at: <https://www.gordonengland.co.uk/hardness/vickers.htm>. Accessed January 19, 2018.

m_0 is the initial mass, m_f is the mass of the sample after thermal cycling and dehydration.

2.5 Flexural strength

Based on the ISO standard, the size of flat specimen is 65mm x 10mm x 3mm, with 20 specimen per group. In order to mimic the clinical setting, a group of curved specimen were prepared. The length between the two ends of the curved sample is 65mm with 50mm between the lowest points of two curves. The thickness and width of the specimen are 2.6mm and 20 mm.

$$\text{Flexural Strength: } FS = 3Pl/2bd^2$$

P being the load at the fracture of the sample, l being the distance between the supports, b being the width of the sample, and d being the thickness of the sample. In this experiment, $l = 50.0\text{mm}$, $b = 10.0\text{mm}$, and $d = 3.0\text{mm}$. Two sets of specimen, with ten specimen in each set, will be manufactured for this test. One set of specimen will be processed by the traditional method and another set of specimen will be processed by the new method. All of the specimen will be stored in distilled water at 37°C for 24 hours before test. The load at the fracture of the specimen will be measured and recorded using an Instron universal tester (Figure 2.5).

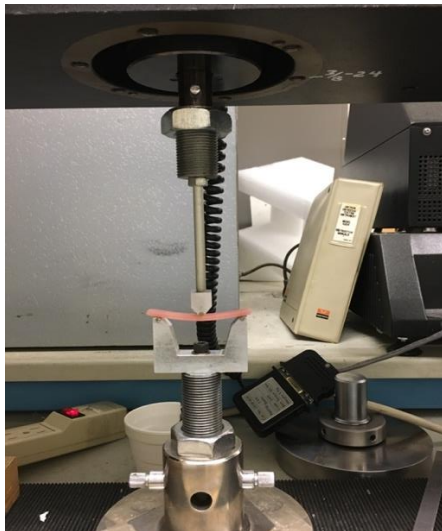


Figure 2.5 Three points bending test using Instron

2.6 Color stability

The specimen were shaped into round discs with a 10 mm diameter and a 2mm thickness. The specimen were immersed in distilled water for 24 hours before the measurement. The color was measured by the photoradiometer (380nm-780nm) (Figure 2.6) using $L^*a^*b^*$ color space (Figure 2.7). Measurements that were taken using a gray backing were used to analyze color. Measurements that were taken using a white and black backing are used to analyze translucency. After the first measurement, the specimen were divided into three groups. Each group contained both experimental group specimen and control group specimen. There are 5 specimen in the control groups and 5 specimen in the experimental groups for coffee and tea. There are 4 specimen in the control groups and 4 specimen in experimental groups for Coca-cola. The three groups were immersed in coffee, tea, and Coca-cola respectively. The temperature of tea and coffee were kept at 60 °C, the coke was set at 23°C. The second and third measurements were taken after 7 days, 14 days and 28 days. Please refer to the appendix for all measured raw data.



Figure 2.6 Photoradio meter

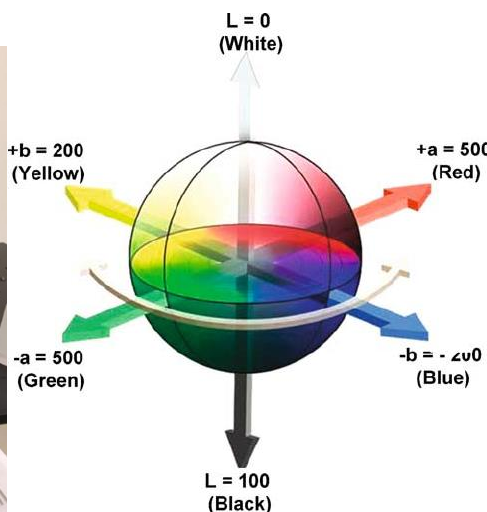


Figure 2.7 $L^*a^*b^*$ Color space²⁷

²⁷ Sudhir KS, The cubical CIE Lab color space.; 2009. Available at: https://www.researchgate.net/publication/23789543_Radiosterilization_of_Fluoroquinolones_and_Cephalosporins_Assessment_

3. Results

3.1 Hardness

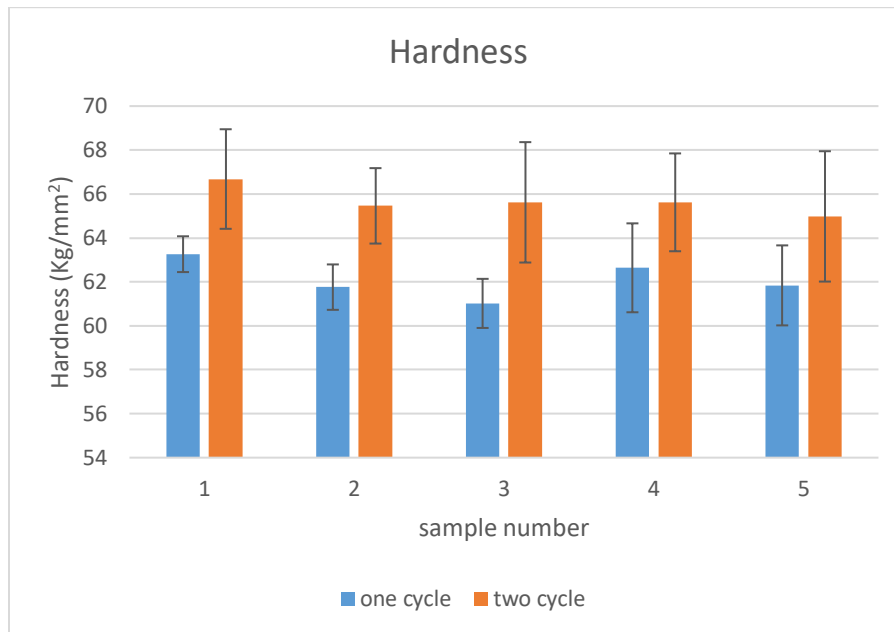


Figure 3.1 Surface Hardness result. In the graph, the blue columns are for one-cycle specimen, orange column is for two-cycle specimen. Each groups contain 5 specimen, and 5 measurement were taken for each sample.

The mean value of the hardness for the control group is 62.1(Kg/mm²), while for the experimental group, the mean value is 65.7 (Kg/mm²). There was a statistically significant difference between groups as determined by one-way ANOVA ($F(1,48) = 42.06, p = 4.52 \times 10^{-8}$). The variance for the two-cycle group is 5.21, which is much higher than that of one-cycle processed group.

3.2 Solubility

The mass lost by two-cycle specimen is 2/3 of the mass lost by the one-cycle specimen.

The mass lost in water is the portion of the non-polymerized material. The result demonstrate that the two-cycle materials have the higher degree of polymerization than the one-cycle materials.

Sample	one cycle rel mass loss(%)	two cycle rel mass loss(%)
1	0.6041	0.2400
2	0.5775	0.5032
3	0.6308	0.5344
4	0.6108	0.5182
5	0.5346	0.5029
6	0.8166	0.5426
7	0.5273	
8	0.5424	
AVG	0.6064±0.093	0.4736±0.11

Figure 3.2 Comparison between the mass loss.

The mean relative mass loss values between one-cycle and two cycle method were 0.60 and 0.47 respectively. The variance of experimental group is less than four times of the variance of the control group; it is reasonable to consider they have equal variance. From result of the t-test assuming equal variance (with 95% confidence), $P=0.0356$.

3.3 Flexural Strength

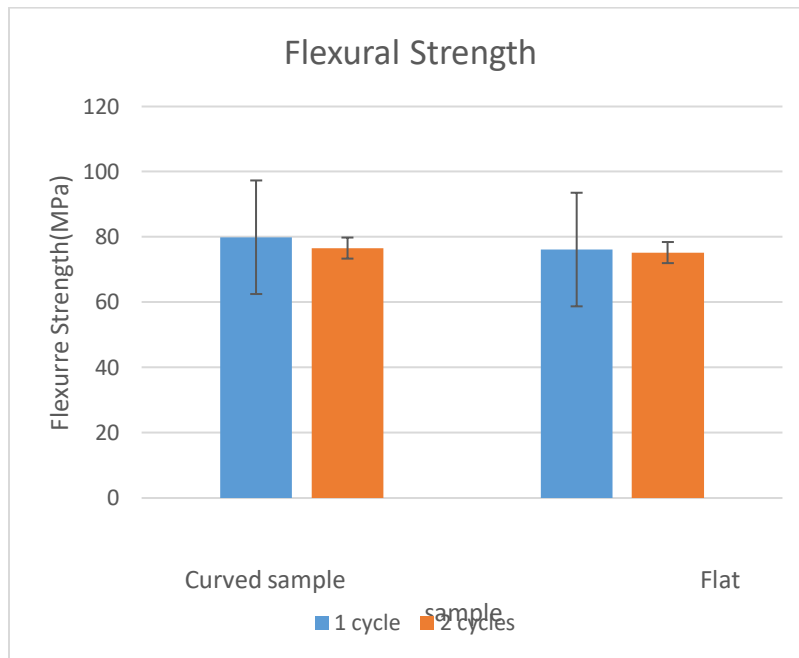


Figure 3.3 Comparison between the flexural strength of flat sample. The blue columns represent the one-cycle method; the orange column represents the two cycle method. The p value is $0.7538 > 0.05$.

3.4 Color stability

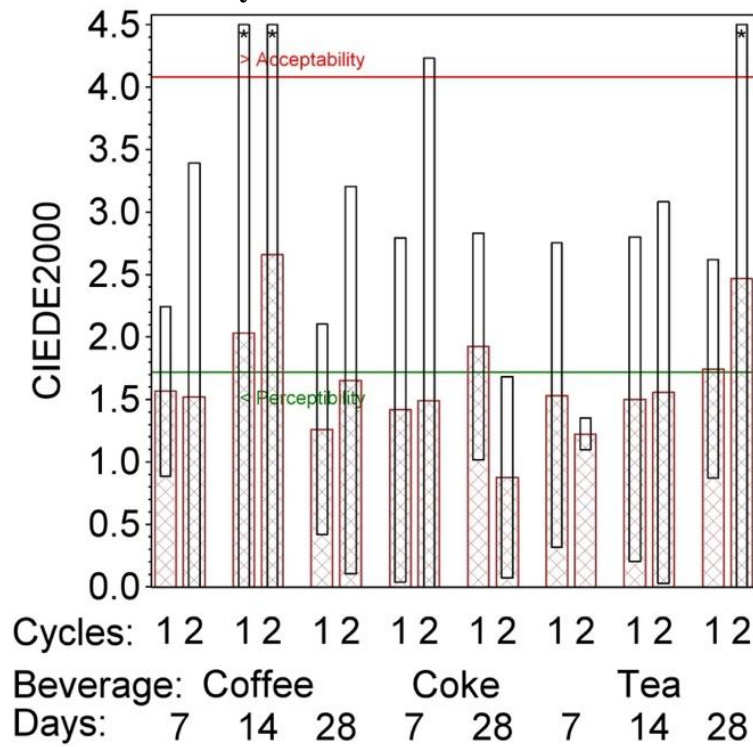


Figure 3.4 Comparison between specimen made by two techniques in susceptibility of color change in three different beverages.

Type 3 Tests of Fixed Effects				
Effect	Num DF	Den DF	F Value	Pr > F
Cycles	1	12.5	0.02	0.8835
Beverage	2	12.5	0.46	0.6431
DaysStained	3	32	11.41	<.0001
Cycles*Beverage	2	12.5	3.79	0.0515
Cycles*DaysStained	3	32	0.40	0.7509
Beverage*DaysStained	5	31.7	1.69	0.1665
Cycles*Beverage*DaysSt	5	31.7	0.17	0.9723

Figure 3.5 Statistically analysis of relative translucency parameter

There is no observable difference in color of two groups. In the coffee and tea solution, the ΔE of two group is nearly equal. In the coke solution, the mean value of ΔE for experiment group is slightly lower than that of the control groups. However, the differences between two group are not statistically significant ($p > 0.05$). Similarly, no significant effect ($p > 0.05$) of cycles was found for the relative translucency parameter.

4. Discussion

The point of interest in this study is whether two-cycle method could impact some of the physical properties of the material. Based on data, the null hypothesis that two-cycle method has no impact on the hardness is rejected ($P < 0.05$). Hardness indicates the degree of resistance of the material when encountering plastic deformation. The surface hardness is related to the material's susceptibility to scratch or wear, which is a type of deformation; a harder material would be more resistant to the scratching or wear. PMMA resins have relatively low surface hardness and are in risk of being damaged by abrasion²⁸, causing accumulation of plaque and compromising color stability and even the longevity of the denture base. The Vickers hardness (VH) test is a frequently used method to evaluate the material's ability to resist penetration of certain load.

Because the degree of polymerization is higher in the 2 cycle group, compared to the control it is expected that the material becomes stiffer and more resistant to penetration²⁹. Unpolymerized monomers will plasticize the denture base and result in lower modulus and hardness. So VH (Viker Hardness) test can be used to indirectly measure the degree of polymerization³⁰. As expected, the specimen processed by 2-cycle technique are harder than the sample produced by the traditional method. The extra heating steps increase the degree of polymerization. When the indenter penetrated the sample, the amount of material displaced in the two-cycled method decreased. The decreased diameter of the indentation caused increase in the VH number. The accuracy of the result might be affected by the subjectivity of research for

28 Harrison Z, Johnson A, Douglas CWI. An in vitro study into the effect of a limited range of denture cleaners on surface hardness and removal of *Candida albicans* from conventional heat-cured acrylic resin denture base material. *J Oral Rehabil* 2004; 31: 460–467.

29 Anusavice KJ. *Philip's Science of Dental Materials*. 11th edn. St. Louis: Elsevier Science, 2003: 140–160.

30 Rueggeberg FA, Craig RG. Correlation of parametric tests used to estimate monomer conversion in a light-cured composite. *J Dent Res* 1988; 67: 932–937

the length of the indentation is observed and determined by human eyes; at the same time, the elastic recovery of the material and the sharpness of the edge of indentation may also affect the result.

PMMA monomers can dissolve in water and may cause potential adverse reactions and biofilm-infection³¹. The soluble portion of the denture base contains: initiator, monomer, and plasticizer. Thus, the solubility of denture base material is related to loss of residual monomer during immersion in water. Usually, the residual monomers are trapped between the polymer chains or micropores in the material³². The aim of this study is to investigate the efficacy of two cycle method in reducing the portion of soluble monomer by elevating the degree of cure. The relative mass loss values between one-cycle and two cycle method was observed to have means of 0.60% and 0.47%. The mass loss for both groups were small because the material used is commercially approved PMMA and the one-cycle process results is a high degree of cure. But it is evident that the mass lost by two-cycle specimen is nearly 2/3 of the mass lost by the one-cycle specimen and the t-test result show there was significant difference between the two means. The result showed that the two-cycle materials have the lower water solubility than one-cycle materials and the null hypothesis was rejected($P < 0.05$). Because two-cycle material has a higher degree of polymerization and less free monomers. This finding indicates denture bases manufactured via new method could cause less irritation to denture-bearing tissues and longer denture base life-span than would denture materials manufactured by the traditional method. However, further research is needed to determine the exact content of the monomer in the denture base.

³¹ Weaver RE, Goebel WM *J Prosthet Dent.* 1980 Feb; 43(2):138-42.

³² Sideridou I, Tserki V and Papanastasiou G, *Biomaterials* **24**: 655–665 (2003).

Discoloration of denture base indicates aging and potential damage of dental material. Color change of denture base also compromises the esthetic quality of the material. Therefore, regardless of the processing technique used, the denture base should be evaluated on its color stability. If the degree of polymerization is higher, it is expected that less water sorption will take place and the denture base will resist staining. The discoloration is related to absorption of colorant in the beverage. Thermocycling, which could artificially enhance the water sorption of the denture base, was not used in this study. That could explain why the color change in coffee, which is a strong staining component³³, was not noticeable. The method may also account for the minor change in color, whereas the results from Dr. Alp's study, where thermocycling in coffee was used, the discoloration was insignificant³⁴. For Coca-Cola, the major colorant 4-methylimidazole is less polar, which may account for the poor staining. The major colorant in tea, tannin, is hydrophilic and an increased trend of ΔE was observed. Denture produced by new method had slight higher ΔE , but the difference is not statistically significant. Overall in this study, most specimen that were immersed in colored solution did not show any perceivable color change. For specimen that had a perceivable color change, the discoloration was below the acceptability line; which means that change is not significant. The data showed that processing method had no significant effect on the color of the PMMA denture base, ($P>0.05$); thus the null hypothesis was not rejected. For the transparency, the P value is 0.0515 which is greater but close to the threshold of significance. The results from color stability could not disapprove the null hypothesis that the new technique does not affect the color stability of dental prostheses. Due to the sample size, the error bar is relatively high and it is possible that when more specimen

³³ Imirzalioglu P, Karacaer O, Yilmaz B, Ozmen I. Color stability of denture acrylic resins and a soft lining material against tea, coffee, and nicotine. J Prosthodont 2010;19:118-24.

³⁴ Alp G. Optical properties and surface roughness of prepolymerized CAD-CAM poly(methyl methacrylate) denture base materials. In press.

were tested, the P value for transparency will change; thus, further research on transparency is necessary.

Flexural strength is a crucial factor which is correlated to the longevity of the denture. Low flexural strength is positively related to the frequency of fracture and breakage³⁵. The flexural strength of the denture base is affected by many factors, including level of polymerization³⁶. The data failed to reject the null hypothesis($p>0.05$) using the ISO standard sample. One possible explanation is that the shape of the sample affected the measured change in flexural strength. In the clinic setting, the denture base could never be flat and straight. When absorbing water, the denture base tends to expand along the curve. However, the specimen are in a rectangular shape, they expanded along a straight line. It is possible that the internal strain and stress accumulate differently on a linear and curvilinear configuration. Therefore, the result from testing a curved sample is necessary. The ANOVA result from curved sample failed to reject that the processing method have no significant impact on flexural strength ($F(1,18) = 0.0511$, $p = 0.824$). Thus, even as the content of polymerized material increased, as proved by solubility test, the change in flexural strength was not evident.

5. Conclusion

Within the scope of this study, following conclusions were drawn:

1. Two-cycle processed PMMA materials had relative relatively higher hardness than the one-cycle processed PMMA materials.
2. Two-cycle processed PMMA materials contained less soluble content; therefore, the

³⁵ SHARMA P, GARG S, KALRA N. Effect of Denture Cleansers on Surface Roughness and Flexural Strength of Heat Cure Denture Base Resin-An In vitro Study. *Journal Of Clinical & Diagnostic Research* [serial online]. August 2017;11(8):94-97.

³⁶ Jagger RG. Effect of the curing cycle on some properties of a polymethylmethacrylate denture base material. *J Oral Rehabil.* 1978;5:51-57.

degree of polymerization of the two-cycle processed PMMA was higher.

3. No significant difference was found in the color change with conventional processed PMMA and PMMA treated with two-cycle method.
4. No significant difference was found in the flexural strength with conventional processed PMMA and PMMA treated with two-cycle method.

6. Recommendation

Additional studies may be conducted to investigate the effect of processing method on surface roughness and water sorption, which are also important properties that related to the quality of the denture base material. A more balanced design should be developed for the color stability test described in this thesis. In addition, the effect of using distilled water instead of artificial saliva when preparing specimen should be considered. The chemistry of artificial saliva could affect the structure and monomer content in the sample. More beverages would be used in color stability test. The effect of thermocycling(aging) on the flexural strength of the material could also be studied. Finally, long-time testing should be conducted to determine the degree of resistance of the material toward aging.

7.Appendix

Column1	one cycle	two cycle
1	62.4	64.9
1	62.9	64.2
1	62.9	66.3
1	64.5	69.4
1	63.6	68.6
average	63.26	66.68
2	61.5	65.6
2	62.7	67.8
2	62.7	64.8
2	61.7	66
2	60.2	63.1
average	61.76	65.46
3	60	68.6
3	62	65
3	59.7	66.9
3	61.3	66.3
3	62.1	61.3
average	61.02	65.62
4	61	67.3
4	63.5	68.1
4	60	63.8
4	64.6	62.9
4	64.1	66
average	62.64	65.62
5	63.8	65.3
5	61.3	63.6
5	59.4	69.4
5	63.5	65.3
5	61.2	61.3
average	61.84	64.98

Table 7.1 Vicker Hardness measurements

sample	before(g)	after(g)	differences	relative mass loss
1	0.27147	0.26983	0.00164	0.604118319
2	0.25799	0.2565	0.00149	0.577541765
3	0.27427	0.27254	0.00173	0.630765304
4	0.25705	0.25548	0.00157	0.610776114
5	0.24878	0.24745	0.00133	0.534608891
6	0.2633	0.26115	0.00215	0.816559058
7	0.2579	0.25654	0.00136	0.527336177
8	0.26181	0.26039	0.00142	0.54237806
AVG	0.26157125	0.259985	0.00158625	0.606431326

Table 7.2 Raw data for solubility test of one-cycle method

sample	before(g)	after(g)	differences(g)	relative mass loss(%)
1	0.25412	0.25351	0.00061	0.240044074
2	0.24839	0.24714	0.00125	0.503240871
3	0.25448	0.25312	0.00136	0.534423137
4	0.25857	0.25723	0.00134	0.518234907
5	0.27242	0.27105	0.00137	0.502899934
6	0.26353	0.2621	0.00143	0.542632717
AVG	0.258585	0.257358333	0.001226667	0.473579273

Table 7.3 Raw data for solubility test of two-cycle method

cycle	specimen	F(N)	L(mm)	b(mm)	h(mm)	flexture strength
1	1	102.4	50	10	3.122	78.79441266
1	2	102.7	50	10	3.124	78.92410334
1	3	100.22	50	10	3.2	73.40332031
1	4	97.19	50	10	3.154	73.27561422
1	5	109.29	50	10	3.16	82.08570341
1	6	80.37	50	10	3.116	62.08123298
1	7	97.46	50	10	3.11	75.57303998
1	8	86.25	50	10	3.104	67.13932056
1	9	103.9	50	10	3.118	80.15388723
1	10	100.94	50	10	3.062	80.74473082
1	11	98.59	50	10	3.195	72.43565724
1	12	91.67	50	10	3.04	74.39458535
1	13	95.87	50	10	3.104	74.62778739
1	14	65.66	50	10	3.164	49.19140105
1	15	93.37	50	10	3.078	73.91479746
1	16	96.43	50	10	3.037	78.41223744
1	17	87.24	50	10	2.968	74.27610596
1	18	90.96	50	10	3.052	73.23904227
1	19	96.46	50	10	3.035	78.54004218
1	20	97.46	50	10	3.081	77.0024022
1	21	84.82	50	10	2.993	71.01434699
2	1	92.46	50	10	3.157	69.57704641
2	2	103.37	50	10	3.066	82.47294039
2	3	112.68	50	10	3.196	82.73600762
2	4	102.49	50	10	3.185	75.77464025
2	5	89.77	50	10	2.998	74.90817761
2	6	101.76	50	10	3.155	76.67250183
2	7	90.87	50	10	3.054	73.07077709
2	8	95.03	50	10	3.064	75.91794971
2	9	87.48	50	10	3.049	70.57569913
2	10	84.54	50	10	3.054	67.98067014
2	11	91.66	50	10	3.154	69.10631546
2	12	60.23	50	10	2.958	51.62710674
2	13	80.81	50	10	2.941	70.07067823
2	14	104.21	50	10	3.133	79.62507777
2	15	100.19	50	10	3.155	75.48956327
2	16	69.54	50	10	2.782	67.3878652
2	17	91.13	50	10	3.147	69.01272051

2	18	92.76	50	10	3.026	75.97735248
2	19	100.07	50	10	3.083	78.96199524
2	20	84.83	50	10	2.883	76.54581397
2	21	71.54	50	10	2.893	64.10816291
2	22	88.08	50	10	2.978	74.48849206
2	23	104.17	50	10	3.166	77.94389536
2	24	99.3	50	10	3.137	75.68006118

Table 7.4 Raw data for flexural strength test for rectangular specimen

cycle	specimen	F(N)	l(mm)	b(mm)	h(mm)	flexture strength
1	1	162.1	50	20.1	2.619	88.1814476
1	2	186.42	50	20.561	2.601	100.5145261
1	3	126.99	50	20.464	2.683	64.65452332
1	4	124.61	50	20.174	2.642	66.36766166
1	5	145.76	50	19.907	2.554	84.18835442
1	6	164.16	50	19.934	2.637	88.82063901
1	7	169.52	50	19.768	2.568	97.52802642
1	8	144.4	50	19.679	2.511	87.28347039
1	9	130.88	50	19.855	2.569	74.90942264
1	10	200	50	20.431	2.624	106.6286655
2	1	111.5	50	20.791	2.652	57.18915938
2	2	157.79	50	20.439	2.563	88.14219947
2	3	177.93	50	19.756	2.6	99.92283072
2	4	110.83	50	20.054	2.603	61.17433398
2	5	164.77	50	19.971	2.537	96.13879159
2	6	177.39	50	19.335	2.558	105.1586835
2	7	173.07	50	20.003	2.612	95.1133716
2	8	129.12	50	19.624	2.516	77.95535831
2	9	187.09	50	20.073	2.598	103.5669676
2	10	145.09	50	20.043	2.451	90.37522575

Table 7.5 Raw data for flexural strength test for curved specimen

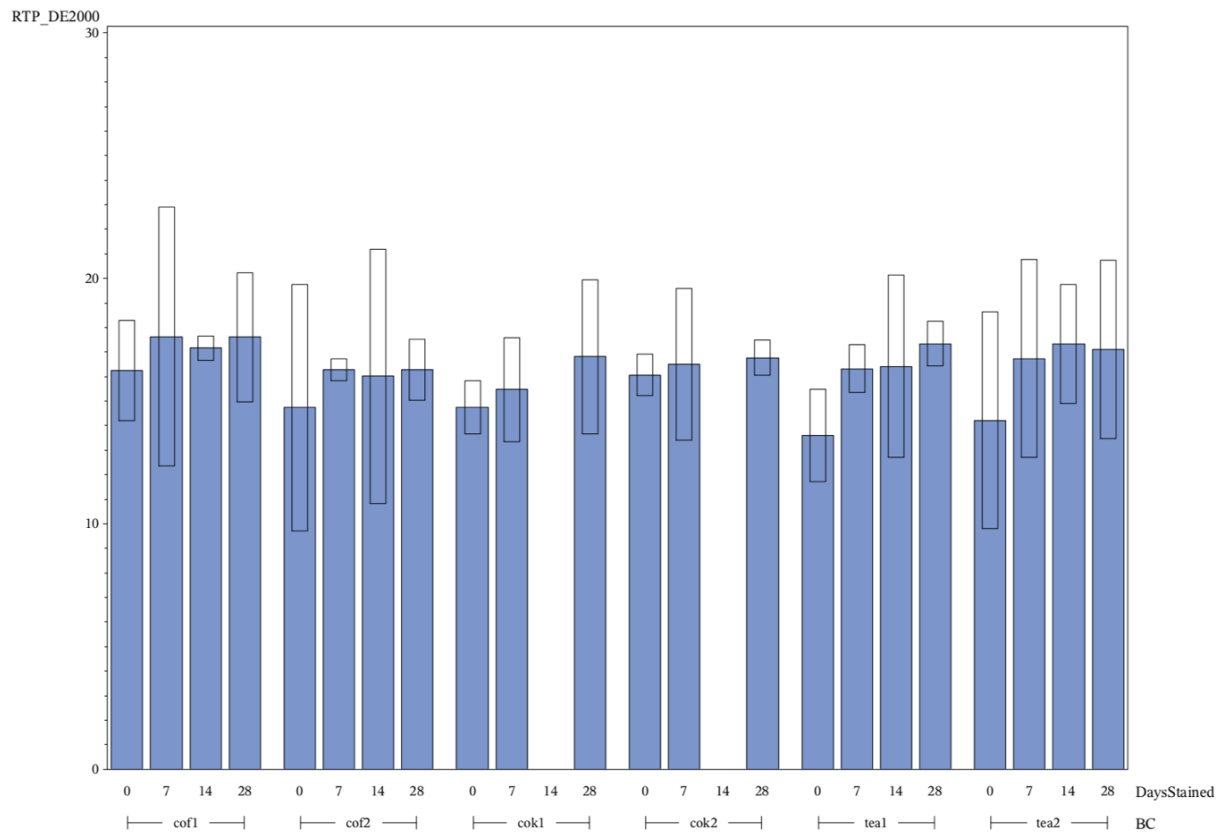


Figure 7.1 Relative transparency parameter graph for color stability